

## Preparation and Characterization of Activated Carbon from *Pandanus candelabrum* Stem

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### ABSTRACT

*Pandanus candelabrum* stem, a new precursor, was used to synthesise activate carbon. The effect of sodium hydroxide, NaOH, zinc (II) chloride, ZnCl<sub>2</sub> and phosphoric acid, H<sub>3</sub>PO<sub>4</sub>, different agents on prepared adsorbents was investigated. The adsorbents were prepared with chemical agents and carbonized at 400 °C for 1 hour. Surface morphology, elemental composition and functional groups were analysed with scanning electron microscopy (SEM), energy dispersive X-ray (EDX), X-ray diffraction (XRD) and fourier transform infrared spectroscopy (FTIR), respectively. The image analysis showed the presence of both micropores and mesopores in the adsorbents. The H<sub>3</sub>PO<sub>4</sub> activated carbon had the maximum surface area (2648 m<sup>2</sup>/g), pore volume (1.683 cm<sup>3</sup>/g) and highest adsorption for iodine and methylene blue were 541 and 105 mg/g. *Pandanus candelabrum* stem is an alternative material that can be used to synthesis high porous adsorbent because is abundant, easy to access, inexpensive and readily available.

Keywords: Activated, adsorption, iodine number, methylene blue, *Pandanus candelabrum*, reagents

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### INTRODUCTION

The most widely used adsorbent is activated carbon, which has high surface area, adsorptive capacity and is inexpensive (Sahira *et al.*, 2013; Yorgun & Yildiz, 2015; Kumar & Jena, 2017). Biomass is a natural carbon source which is used for the preparation of green adsorbents, which have several advantages over non-renewable sources. This renewable biomass is abundant, cheap, environmentally friendly and renewable (Rai *et al.*, 2016). The application of biomass wastes for the synthesis of solid adsorbent reduces the impact of environmental pollution (Cadavid *et al.*, 2016). One of the popular methods employed in the preparation of activated carbon is the chemical activation process (Zibrik *et al.*, 2017). This method entails doping of carbon source with activating reagent, followed by carbonization at chosen temperature values. This will have a significant effect on the carbon matrix morphology resulting to changes in the pore structure arrangement (Nafsun *et al.*, 2020). Which is primarily, due to interaction between carbon atoms and chemical agents such as strong acid (H<sub>3</sub>PO<sub>4</sub>),

base (NaOH) or salts (ZnCl<sub>2</sub>) (Gu & Wan, 2013; Shamsuddin *et al.*, 2016). The chemical method exhibits a very short time and low temperature during pyrolysis process (Giraldo *et al.*, 2018). These advantages have resulted to the production of affordable and cheaper adsorbents using chemical activation method. Recently, many researchers have use different renewable resources for the preparation of adsorbent, such as mango tree bark (Dim, 2013), *Malva sylvestris* (Ramavandi & Asgari, 2018), fennel seed (Hussein & Jasim, 2019) and kaolinite clay (Dim *et al.*, 2020).

*Pandanus* stem is an agricultural by product which is mainly composed of cellulose, hemicellulose, and lignin. Therefore, *Pandanus* stem will be a suitable lignocellulosic biomass for preparation of porous active carbons. *Pandanus candelabrum* originated from *Pandanus* plant (Screw pine), which belongs to the family Pandanaceae which comprises about 700 species and is widely distributed in tropical and subtropical regions (Fillaeli *et al.*, 2019). They are found in the regions of South-East Asia, Pacific islands,

Madagascar, Indian Ocean islands, India and West Africa. It is a dioecious, small tree up to 12 m tall with branched trunk and aerial roots (Mario & Hiromatsu, 2018). *Pandanus* tree is a multi-purpose plant with different uses. It is known locally as screw pine. The leaves are used for making mats, baskets and fish traps, while the fibrous root is used for brushes (Akpabio & Akpakpan, 2012). In traditional medicine the leaves are used for the treatment of sore throat, and bark infusions are used to cure diarrhoea, dysentery, enteritis, antispasmodic, diuretic, and stimulant properties (Baba *et al.*, 2016). Despite of its various applications there is none for using *P. candelabrum* stem for synthesis of active adsorbent.

This work used *P. candelabrum* stem as carbon source to produce active carbon with  $H_3PO_4$ ,  $ZnCl_2$  and NaOH. Based on literature there is no study on activated carbons synthesized from *P. candelabrum* stem. The adsorbents were characterized in terms of functional group, elemental composition and morphology.

## MATERIALS AND METHODS

### Materials

*Pandanus candelabrum* stem, which was used as a precursor was obtained from Ogbunka, Anambra State, Nigeria. To remove impurities distilled water was used to wash the sample severally, after which it was dried for 6 h at 80 °C. Chemical agents used were  $H_3PO_4$ ,  $ZnCl_2$  and NaOH.

### Preparation of Adsorbent

Accurately weighed 15 g of pulverized *Pandanus* stem was impregnated with activating agents in a ratio of 1:4 (w/w)  $H_3PO_4$ , (Yorgun and Yildiz, 2015) 1:3 (w/w)  $ZnCl_2$  and 1:4 (w/w) NaOH (Kilic *et al.*, 2012) (Precursor: Agent) by weight for 12 h. To ensure proper mixing and penetration of the agents into the internal structure of precursor the doping was done at 80 °C for 6 h. After impregnation, mixtures were carbonized at 400 °C (Yorgun & Yildiz, 2015) in nitrogen flow (200  $cm^3/min$ ) at heating rate of 10 °C/min, for 1 h in a horizontal tube furnace (model Y02PB, Thermocraft, Inc, USA), and was cool by open air. The resulting products were neutralized to pH of 7,

by washing it several times with distilled water, and was oven dried at 105 °C for 12 h. The product was pulverized to sizes of 0.1 – 0.2 mm.

The modified adsorbents were labelled as sodium hydroxide activated carbon (SAC), zinc chloride activated carbon (ZAC) and phosphoric acid activated carbon (PAC) and stored in a tight container for further use. A control sample was prepared without impregnation and was labelled as control activated carbon (CAC) and stored for further used.

### Characterization of Adsorbents

The physicochemical properties of the *P. candelabrum* stem as performed using the elemental analyzer and American Standard for Testing Materials, standard test methods (Yorgun & Yildiz, 2015, Shamsuddin *et al.*, 2016). Scanning electron microscope (SEM)-energy dispersive X-ray (EDX), (SEM-EDX, JEOLJSM 7600F) was used to determine the morphology and elemental composition. Thermo Electron Nicolet 4700 FTIR spectrometer recorded the spectra of the adsorbent from 4000 to 500  $cm^{-1}$  resolution. The Brunauer-Emmett Teller (BET) method was used to determine specific surface area and pore volume using a micromeritics ASAP 2020. Methylene blue (MB) and iodine were used to test the adsorption capacities of adsorbents. For MB, about 250 mg of adsorbents were mixed with 25 mL of 1.5 g/L MB standard solution and shaken at 250 rpm in a thermostatic shaker at 25 °C for 24 h (Liu *et al.*, 2019). While iodine value was tested by mixing 250 mg of adsorbents with 25 mL of 0.10N iodine solution, and was shaken for 30 min, and then iodine adsorption amount was determined by titration against standard  $Na_2S_2O_3$  (Liu *et al.*, 2019). The MB and iodine adsorption amount of AC was determined according to Sahira *et al.*, (2013), Hassan *et al.*, (2014) and Liu *et al.*, (2019).

## RESULTS & DISCUSSION

### Characterization of Adsorbents

The result of characterisation of *P. candelabrum* stem are presented in Table 1. The stem was found to have moisture content of 4.99%, ash content of 6.32%, which was low, and volatile matter of 72.3%, which was high. As seen from Table 1, the

**Table 1.** The properties of *Pandanus candelabrum* stem

| Proximate analysis (%) |       | Ultimate Analysis (%) |       |
|------------------------|-------|-----------------------|-------|
| Fixed carbon           | 12.00 | C                     | 45.04 |
| Moisture content       | 4.99  | H                     | 6.72  |
| Ash content            | 6.32  | N                     | 0.42  |
| Volatile matter        | 72.30 | O                     | 43.85 |

**Table 2.** Physicochemical properties of adsorbent

| Parameter                         | CAC    | SAC    | ZAC    | PAC    |
|-----------------------------------|--------|--------|--------|--------|
| Ash content (%)                   | 11.663 | 17.622 | 11.554 | 13.095 |
| Moisture Content (%)              | 7.784  | 4.382  | 0.994  | 0.994  |
| Bulk density (g/cm <sup>3</sup> ) | 0.354  | 0.444  | 0.259  | 0.316  |

high volatile matter and low ash content of *P. candelabrum* stem has made it suitable as an alternative starting material for production of activated carbon.

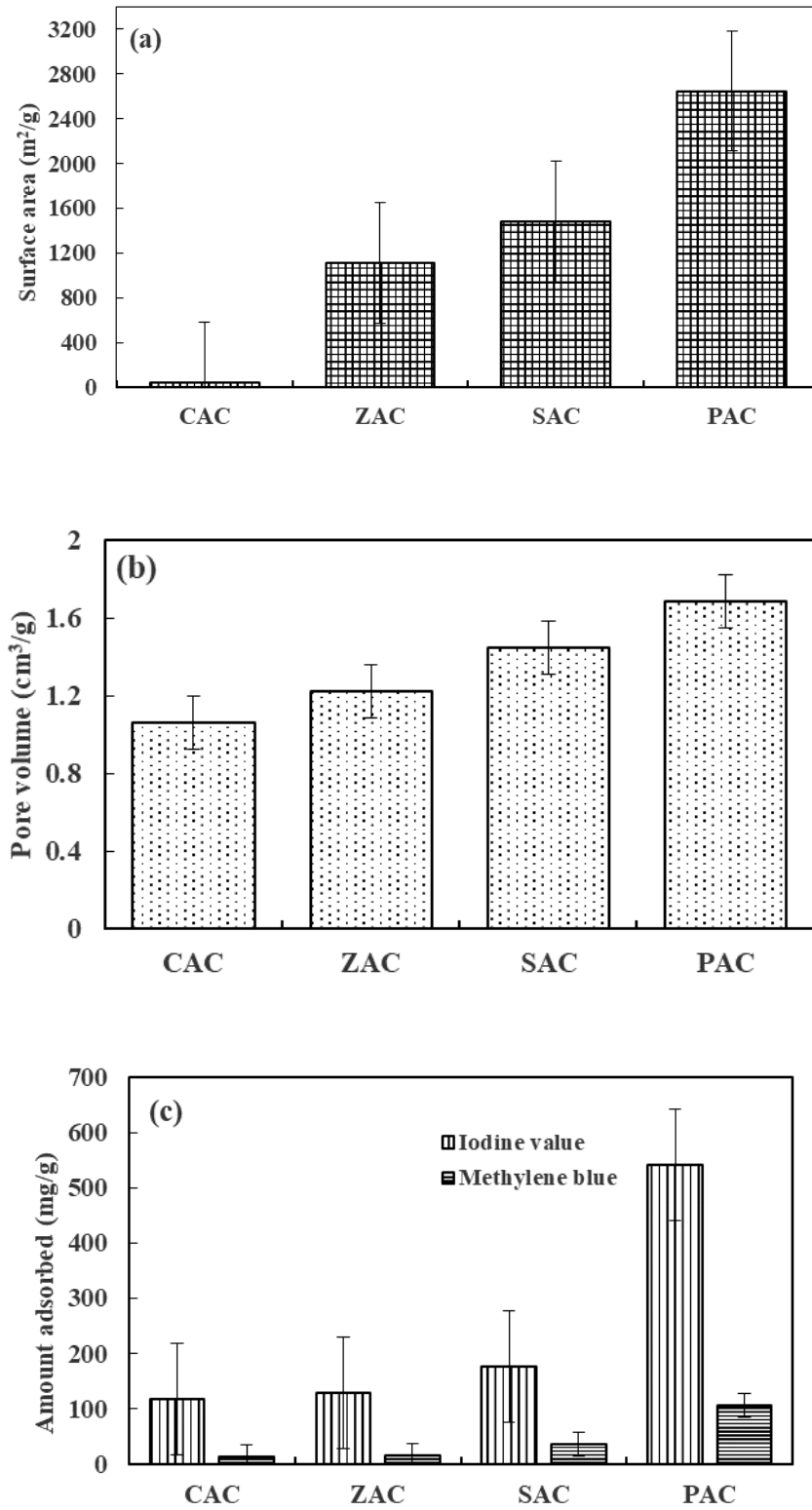
The results of adsorbent characterization are presented in Table 2. The activated carbon produced from *Pandanus* stem with different agents had different properties. The SAC had the highest ash content (17.62), bulk density (0.444 g/cm<sup>3</sup>) and low pore volume (1.220), while ZAC has the lowest bulk density (0.259 g/cm<sup>3</sup>) and ash content (11.554%). The increase in bulk density is because of the decrease in pore volume of the activated carbons which resulted to increase in weight of the adsorbents. The presence of low volatile content may have contributed to the formation of large quantity of non-volatile matter such as ash (Kilic *et al.*, 2012). The properties of adsorbent obtained are in comparison with previous findings by some researchers (Ademulyi *et al.*, 2016).

Figure 1 shows the surface areas, pore volumes and amount of adsorbed methylene blue and iodine for adsorbents activated with different chemical agents. The surface area is 2648 m<sup>2</sup>/g for PAC, 1482.9 m<sup>2</sup>/g for SAC, 1114.13 m<sup>2</sup>/g for ZAC and 43.22 m<sup>2</sup>/g for CAC. Pore volume is 1.683 cm<sup>3</sup>/g for PAC, 1.448 cm<sup>3</sup>/g for SAC, 1.220 cm<sup>3</sup>/g for ZAC and 1.046 cm<sup>3</sup>/g for CAC. In Figure 1, surface area (Figure 1a) and pore volume (Figure 1b), phosphoric had the highest surface area and pore volume, which is 2648 m<sup>2</sup>/g and 1.683 cm<sup>3</sup>/g respectively. The highest surface area of PAC may be due to H<sub>3</sub>PO<sub>4</sub> interaction decomposed some component in the carbon source, such as aliphatic

and aromatic compounds, and this resulted in easy gasification of volatile matters (Kilic *et al.*, 2012). The trends therefore, imply that the accessible area of adsorbent available for methylene blue adsorption follows the decreasing order, PAC > ZAC > SAC > CAC. The properties of adsorbents will favour adsorption.

From Figure 1c, the amount of iodine value and methylene blue adsorbed, is 541 and 105 mg/g for PAC, 175 and 36 mg/g for SAC, 128 and 16 mg/g for ZAC and 118 and 13 mg/g for CAC respectively. It can be observed that adsorption of iodine show increasing in adsorption capacity in the following decreasing order: PAC > SAC > ZAC > CAC. This shows capability of each adsorbent to adsorb small sized atom such as iodine (Nunes *et al.*, 2011). In addition, it can be seen from Figure 1c, PAC had the maximum iodine value and methylene blue (541 mg/g of carbon and 105 mg/g). H<sub>3</sub>PO<sub>4</sub> aids in the degradation of carbon sources as well as in the creation of structures that exhibit cross-linking properties (Budinova *et al.*, 2006). And this will lead to the development of pores of micro and meso sizes in the adsorbent (Danish *et al.*, 2014, Yorgun & Yildiz, 2015).

The highest iodine value by PAC may be due to the presence of large micropores in the adsorbent (Moreno-Virgen *et al.*, 2012). This is followed by SAC which has iodine value of 175 mg/g. Here both micro porosity and mesoporosity were created through segregation and decomposition of graphite layers due to reduction and oxidative transformation. This aids volatilization, that disintegrates the matrix resulting to increased surface area available for adsorption (Foo &



**Figure 1.** Surface areas (a), pore volumes (b) and amount of adsorbed methylene blue and iodine value (c) for carbon activated with different chemical agents

Hameed, 2012). While ZAC and CAC have iodine value of 128 and 118 mg/g of carbon respectively. In Figure 1c, it can also be observed that the adsorbents show the adsorption ability decreased in the following order: PAC > SAC > ZAC > CAC. In this context the ability to adsorb methylene blue shows how each activated carbon will adsorb large molecules such as methylene blue which is much larger than iodine molecules (Cleiton *et al.*, 2011). PAC impregnated with H<sub>3</sub>PO<sub>4</sub> has the highest adsorption value of 105 mg/g for methylene blue. This suggests it has well-developed mesopores and some portions of micropores (Shrestha *et al.*, 2012).

### Scanning Electron Microscope (SEM) Analysis

The micrograph for unmodified carbon (CAC) (Figure 2a) shows broad surface which consists of few pores, aggregates of small particles, with irregular shape, relatively rough surface, and without cavities. The micrographs of modified carbon (Figures 2b-d) of adsorbent particles showed cavities, pores and rough surfaces. There is more presence of pores with openings in the adsorbents synthesized with H<sub>3</sub>PO<sub>4</sub>, NaOH and ZnCl<sub>2</sub> agents. The features on carbon surface revealed clearly the effect of activating agent on the textural properties. The pore openings are due to extraction of some materials from the surface by activating agents (Shamsuddin *et al.*, 2015). The morphological features on the surface of adsorbent are the evidence of the effect of activating agents. PAC activated carbon impregnated with H<sub>3</sub>PO<sub>4</sub> (Figure 2b) has more open pores and cavities. SAC and ZAC activated carbon (Figures 2c-d) also show presence of open pores. The various chemical agents lead to different reaction mechanisms which resulted to adsorbent with different pore structures. The mechanisms by which H<sub>3</sub>PO<sub>4</sub> activates carbon source entails structural degradation and volatilization, whereas activation with NaOH and ZnCl<sub>2</sub> aids the creation of porosity by the expulsion of water molecules present in the carbon precursor (Kilic *et al.*, 2012).

### Energy Dispersive X-ray Analysis

The unmodified carbon (CAC) and modified PAC, SAC and ZAC were evaluated using EDX (Table 3). The analysis showed that carbon content increased from 82.01 to 86.38 %, in the order of CAC < ZAC < SAC < PAC.

The decrease in oxygen content was from 4.06 % to 2.32 %, the trend was in the order of CAC < SAC < ZAC < PAC. But it was observed that SAC had the highest oxygen content among the modified activated carbon. This could suggest that NaOH-activated carbon has functional groups which contain more oxygen atom. SAC has the highest oxygen content of 3.79 % among all the chemical activated carbon. The high oxygen content of NaOH-activated carbon could be attributed to the oxidation of carbon precursor in alkaline environment (Huang & Zhao, 2015). From Table 3, it can also be seen that other elements present in CAC, SAC, ZAC and PAC are calcium, nitrogen, potassium, silicon, phosphorus, aluminium, sulphur, magnesium, sodium and chlorine.

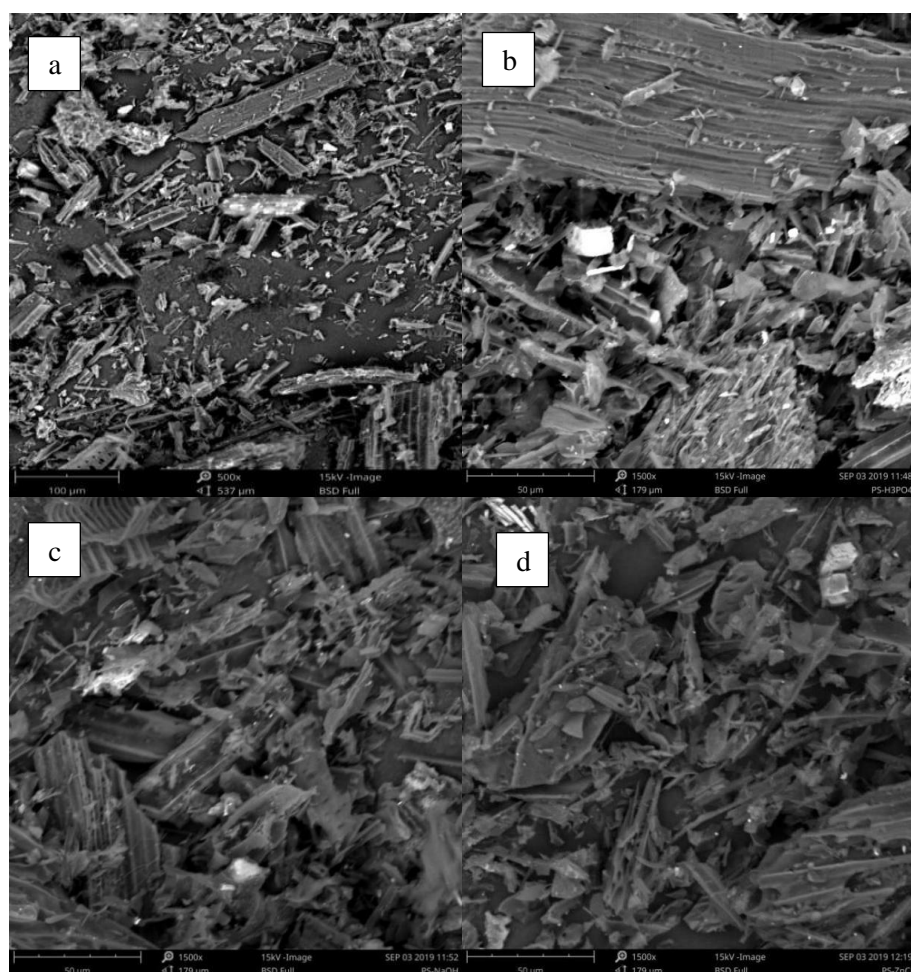
### XRD Analysis

The diffractogram of SAC, ZAC and PAC in comparison with CAC are shown in Figure 3. Activating agents and carbonization caused changes in the crystal structure of the carbons. The CAC displayed peaks at approximately 26.6°, 31.2°, 32.1° and 49.5°, this indicates the presence of graphite (Wang *et al.*, 2009). In comparison with CAC, all the chemically activated carbons show peaks at about 26.7°, 33.1°, 34.2°, 36.6°, 39.2°, 46.8°, 50.2°, 60.1° and 68.3° respectively. The peak intensity changed the activation and subsequent carbonization and this made the peaks look sharper and wider.

This suggests that activated carbons prepared with agents resulted in the formation of micro crystallites which are like graphite (Wang *et al.*, 2011). Therefore, the increase in pore size is because the pore walls are made of hexagonal graphitic microcrystalline which were disintegrated severely. The emergence of new diffractogram pattern shows the creation of new amorphous and crystalline planes (Huang & Zhao, 2015).

### FTIR Surface Analysis

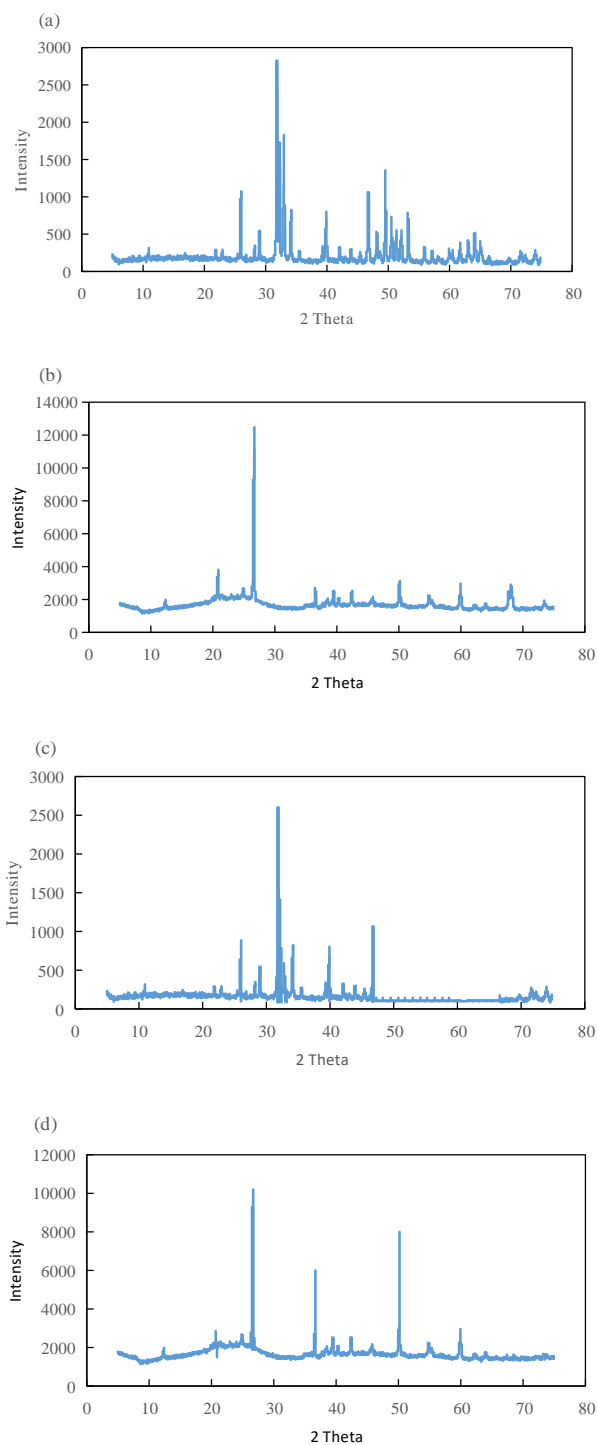
The surface chemical properties of the adsorbents were examined using FTIR analysis. Figure 4 shows the spectrum of the adsorbent obtained without chemical activation (CAC) and the spectra for those obtained with chemical activation (ZAC, SAC and PAC). Similarity as well as difference exist in the absorption pattern of the produced



**Figure 2.** SEM micrograph for activated carbons, (a) Control activated carbon (CAC), (b) Zinc chloride activated carbon (ZAC), (c) sodium hydroxide activated carbon (SAC), (d) Phosphoric acid activated carbon (PAC)

**Table 3.** Elemental analysis of activated carbons

| Element name | Element symbol | Proton number | CAC Wt (%) | ZAC Wt (%) | SAC Wt (%) | PAC Wt (%) |
|--------------|----------------|---------------|------------|------------|------------|------------|
| Carbon       | C              | 6             | 82.01      | 83.10      | 84.42      | 86.38      |
| Oxygen       | O              | 8             | 4.06       | 2.79       | 3.79       | 2.32       |
| Calcium      | Ca             | 20            | 3.22       | 4.20       | 4.12       | 4.02       |
| Nitrogen     | N              | 7             | 1.85       | 0.20       | 1.45       | 1.01       |
| Potassium    | K              | 19            | 1.38       | 0.84       | 0.79       | 1.15       |
| Silicon      | Si             | 14            | 1.35       | 2.21       | 1.63       | 1.52       |
| Phosphorus   | P              | 15            | 1.16       | 1.20       | 1.56       | 2.03       |
| Aluminium    | Al             | 13            | 0.65       | 0.83       | 0.60       | 0.46       |
| Sulphur      | S              | 16            | 0.63       | 0.71       | 0.42       | 0.36       |
| Magnesium    | Mg             | 12            | 0.57       | 0.81       | 0.78       | 0.58       |
| Sodium       | Na             | 11            | 0.13       | 0.55       | 0.45       | 0.18       |
| Chlorine     | Cl             | 17            | 0.00       | 0.63       | 0.00       | 0.00       |



**Figure 3.** XRD for activated carbons: (a) Control activated carbon (CAC), (b) Zinc chloride activated carbon (ZAC), (c) sodium hydroxide activated carbon (SAC), (d) Phosphoric acid activated carbon (PAC)

adsorbents. The weak absorption band at 3900-3600  $\text{cm}^{-1}$  are present in all the carbon spectra. This is assigned to hydroxyl bonds (Yakout & El-Deen, 2016). For CAC, the peak indicates the presence of O-H alcoholic group which was observed at the absorption around 3851  $\text{cm}^{-1}$ . The absorption peak at 2923 and 2853  $\text{cm}^{-1}$  corresponds to aliphatic stretch vibration and C-H functional group (Norouzi *et al.*, 2018). Those weak peaks observed around 2360.52 – 2343.44  $\text{cm}^{-1}$  are assigned to the O-H group showing the presence of phenyl group. While the band observed at 1564  $\text{cm}^{-1}$  indicates C=C bond showing the presence of carbon in aromatic structure (Kilic *et al.*, 2012). The peaks at 1462.40 and 1377.01  $\text{cm}^{-1}$ , correspond to methyl group. The little peak at 1154 - 1250  $\text{cm}^{-1}$  could be ascribed to C-C and C-O stretched vibrations. These positions indicate the presence of acidic, alcohols, phenols, esters and ether group. The peaks at 668 and 721.61  $\text{cm}^{-1}$  indicate the presence of aromatic structures (Wang *et al.*, 2014). This indicates that CAC contains methyl, aromatic and ester bonds which have the presence of oxygen species.

The spectra of PAC, SAC and ZAC indicates reduction in absorption peaks of functional groups. Irrespective of the activating agent used, the spectra were slightly different in the absorption patterns. The weak transmittance bands at 3851-3667  $\text{cm}^{-1}$  exhibited by all the carbons, which is the presence of hydroxyl group. This weak intensity shows that the precursor was dried properly. The bands at 2853, 2923, 1462 and 1377  $\text{cm}^{-1}$  are shrink or reduced for all the modified carbons. While the bands observed at 2343, 2360, 1154, 721 and 668  $\text{cm}^{-1}$  disappeared for all the chemically activated carbon. This absorption illustrates the presence of active carbon in the prepared samples. Furthermore, in PAC, SAC and ZAC the elimination and reduction of some peaks suggests the decomposition and modification of surface chemistry by carbonization and chemical activation. These results suggest that concurrent carbonization and chemical activation caused the formation of new species and decomposition of many bonds that are weak in the functional group (Deng *et al.*, 2010).

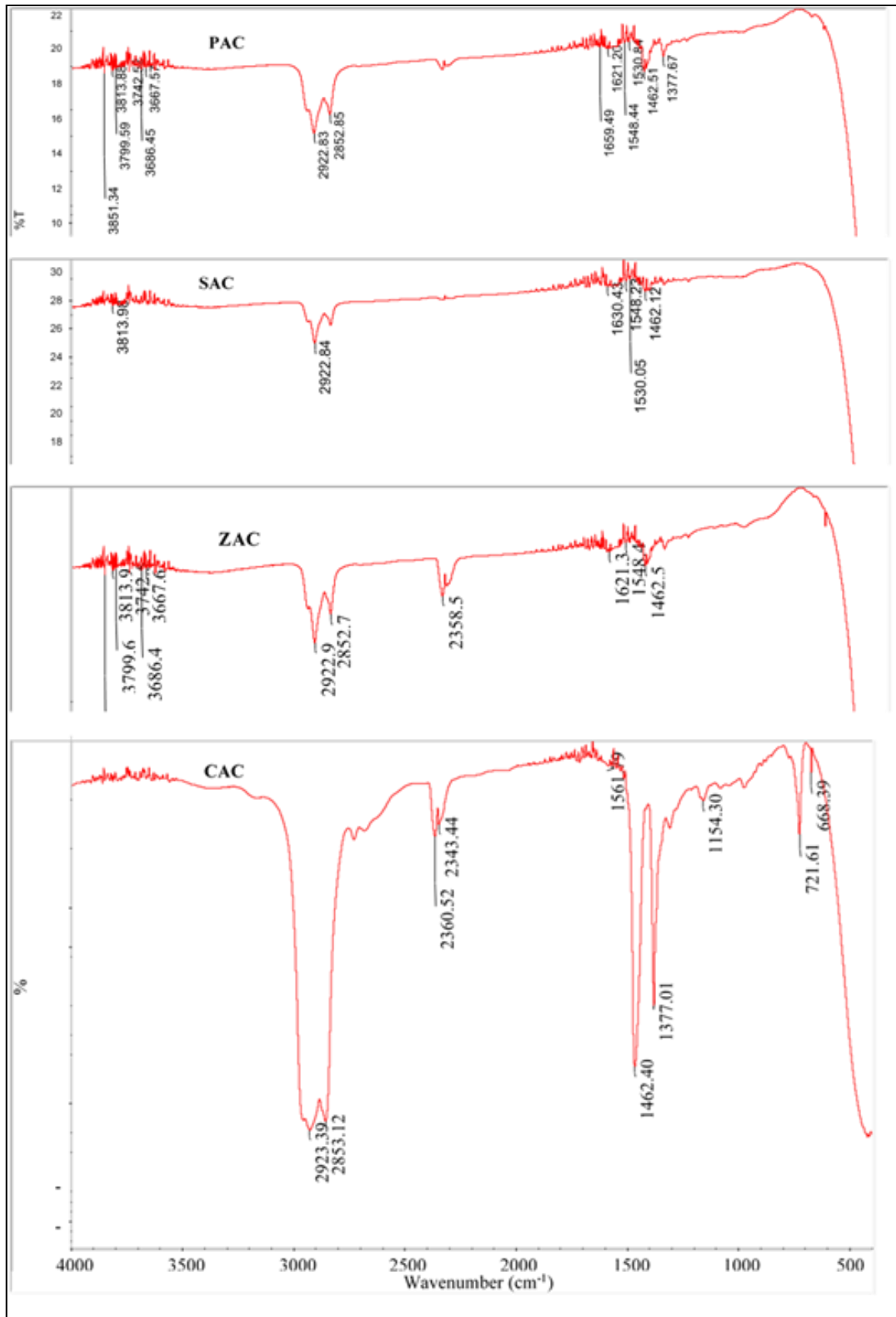


Figure 4. FTIR spectrum of CAC, ZAC, SAC and PAC



## CONCLUSION

*Pandanus candelabrum* stem has been utilized to produce activated adsorbent using different chemical agents. The effect of agents on the properties of the adsorbent were investigated. FTIR results indicate that all the adsorbent prepared has some similarity and differences on the nature of their surface chemical properties. SEM micrograph of adsorbents impregnated with chemical agents showed well-developed pores structure compared to unmodified carbon. The study revealed that chemical agents had effect on the properties of the modified adsorbents. They showed increase in the adsorption of methylene blue in the order of CAC < ZAC < SAC < PAC, and iodine number is in the increasing order of CAC < ZAC < SAC < PAC. This shows that PAC (H<sub>3</sub>PO<sub>4</sub>) activated carbon had the highest iodine number and methylene blue (541 mg/g of carbon and 105 mg/g). The surface area is 2648 m<sup>2</sup>/g for PAC, 1482.9 m<sup>2</sup>/g for SAC, 1114.13 m<sup>2</sup>/g for ZAC and 43.22 m<sup>2</sup>/g for CAC. *Pandanus candelabrum* stem is an alternative lignocellulosic source that can be used to synthesize high porous adsorbent, which is abundant, easy to access, inexpensive and readily available.

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